Analysis of Anionic Contamination on Wafer Surfaces

Thomas Ehmann and Maria Serwe

Semiconductor Industry

Abstract

The quality of a wafer can be seriously affected by ionic impurities on its surface. As part of quality control programs in most semiconductor facilities, anions, cations, metals and amines are routinely measured to determine the level of contamination. Anionic impurities have increasingly attracted attention because they lead to additional etching of the oxide layer, causing light-point defects and affecting the amount of metals that adhere to the wafer surface.

Low sample consumption and cost per analysis together with high speed and short turnover times make capillary electrophoresis (CE) very suitable for the analysis of low ppb levels of anions on wafer surfaces.

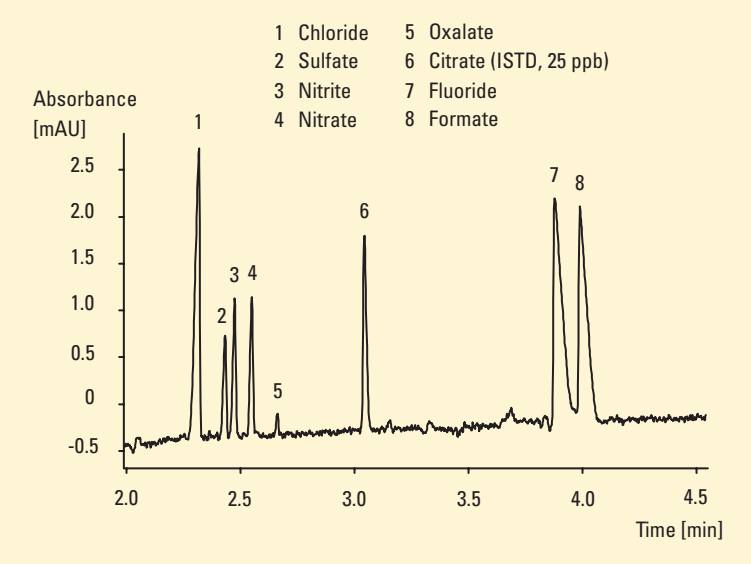

Figure 1
Analysis of anions on wafer surfaces by capillary electrophoresis

Conditions

Sample 500 µl out of 3 ml ultrapure water used for complete wetting of a 200 mm wafer
Injection 125 mbar·s 10 mM NaOH, 5 s water dip followed by -10 kV × 10 s sample injection
Capillary effective length 40 cm
total length 48.5 cm
internal diameter 50 µm
Buffer 2.25 mM pyromellitic acid, 6.5 mM sodium hydroxide, 0.75 mM hexamethoniumhydroxide, 1.6 mM triethanolamine, pH 7.7, buffer replenishment prior to each run
Voltage 30 kV, negative polarity
Temperature 20 °C
Detection signal 350/60 nm reference 245/10 nm
Preconditioning flush with 0.25 M HCl for 0.5 min, flush with buffer for 3 min prior to each run

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Experimental and Results

Anion analysis was performed on a Agilent Capillary Electrophoresis system equipped with diode array detector and controlled through a PC-based Agilent ChemStation. The analysis was based on the Agilent inorganic anion analysis kit (see figure caption for more details). All vials (glass vials for buffer, polypropylene vials for samples) and clear polyurethane caps were washed with ultrapure water (UPW) which fulfilled the requirements of the SEMI guidelines. Anion stock solutions and electrolyte were prepared in a class 10,000 flow box. Calibration standards were prepared daily immediately before use in a clean room under a class 10 laminar flow hood.

Prior to first use new capillaries were treated by a 30 min flush with 0.5 M NaOH, a 30 min flush with electrolyte, and 60 min conditioning at -30 kV. A capillary was considered to be equilibrated when migration time repeatability of 5 consecutive runs using hydrodynamic injection was < 2% RSD.

A typical electropherogram of the analysis is shown in figure 1. Repeatability (n = 20, with internal standard) was < 0.5% RSD for migration times and < 3% RSD for corrected peak areas. Table 1 shows linear ranges and detection limits of the assay using electrokinetic injection. Linearity ranges given are based on correlation coefficients $r^2 > 0.996$.

The data obtained here correlated well with results from ion chromatography (IC). However, CE is more suitable in terms of cost per analysis and ease of use.

Anion	Linear Range (ppb)	Detection Limit (ppb)
Bromide	4–40	0.8 ± 0.5
Chloride	2–18	0.4 ± 0.2
Sulfate	5–48	1.0 ± 0.2
Nitrite	2–23	0.5 ± 0.3
Nitrate	3–31	0.6 ± 0.4
Oxalate	4–44	0.9 ± 0.2
Chlorate	4–42	0.8 ± 0.6
Fluoride	1–10	0.2 ± 0.2
Formate	2–23	0.5 ± 0.5
Phosphate	5–49	0.9 ± 0.1

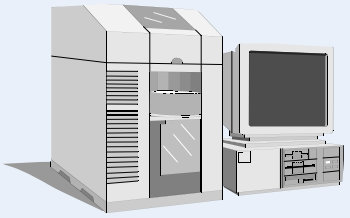
Table 1
Linearity ranges and detection limits of the anions analyzed

Literature

Ehmann et al., “Optimization of the Electrokinetic Sample Introduction in Capillary Electrophoresis for the Ultra Trace Determination of Anions on Silicon Wafer Surfaces”, Chromatographia (1997), Vol. 45, 301–311 (available as Agilent publication number 5966-1991E)

Equipment

Agilent Capillary Electrophoresis system



Thomas Ehmann is a chemist at Wacker Siltronic AG, Central Analytical Laboratories, Burghausen, Germany. Maria Serwe is an application chemist at Agilent Technologies, Waldbronn, Germany.

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